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Resonating torque microbalance for in situ measurements of ferromagnetic films

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Abstract

We describe an instrument for in situ monitoring of the magnetic moment of a thin film during deposition with sub-monolayer sensitivity. The instrument measures the magnetic torque on a film as it is being deposited onto a micro-cantilever excited near its mechanical resonance. Dynamic feedback is used to balance the magnetic torque by applying a mechanical force at the base of the cantilever. © 2002 Elsevier Science B.V. All rights reserved.

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The production and development of many contemporary magnetic thin-film devices require that consistent growth conditions be maintained during film deposition. Typically, film properties are determined ex situ with induction-field ($B-H$) loopers that measure the $M_s t_f$ product for the film, where M_s and t_f are the saturation magnetization and the thickness of the film, respectively. We present here a method based on a micromechanical sensor for making continuous in situ measurements during film deposition. We have developed an instrument that depends on inexpensive, batch-fabricated, micromechanical substrates for quantitative measurements with sub-monolayer magnetic moment sensitivity.

In previous papers [1,2] we describe prototype instruments that demonstrate both the feasibility and some of the pitfalls of in situ magnetometer measurements with micromechanical sensors. For example, it is desirable to measure the magnetic moment of a thin-film continuously during the deposition; however, we found this to be difficult with the prototype instruments due to cantilever bending caused by heating effects during deposition. Also, resonant operation of the cantilever was difficult since the resonant frequency varies with temperature and mass loading. We describe here methods that alleviate these problems. In addition, we have incorporated an optic-fiber interferometer for measuring the motion of the cantilever. Optic-fiber detectors work well in the high noise environment typical of deposition systems.

The resonating torque microbalance is shown schematically in Fig. 1. The optic-fiber interferometer used to measure the deflection of the cantilever is similar to that described in Ref. [3]. A small coil close to the cantilever provides the AC torque field H_T of 300 A/m rms at the resonant frequency (3276 Hz) of the cantilever. H_T is perpendicular to the film and generates a torque due to thin-film shape anisotropy. An oscillator supplies the reference signal for a lock-in amplifier as well as current to the coil through a power amplifier. The cantilever deflection signal from the interferometer is phase shifted, and amplified, and then applied to the cantilever piezoelectric mount. The phase and magnitude of the piezo signal are adjusted to balance the magnetic torque on the cantilever. This process, often referred to as *active damping* [4], alleviates resonant-frequency stability problems associated with temperature drift and mass loading affects. A lock-in amplifier measures the piezo feedback signal that is proportional to the magnetic moment of soft magnetic films. The cantilever is placed between a pair of SmCo permanent magnets that provide a static bias field H_0 of 10 kA/m. Under these conditions, the film should be fully saturated in plane.

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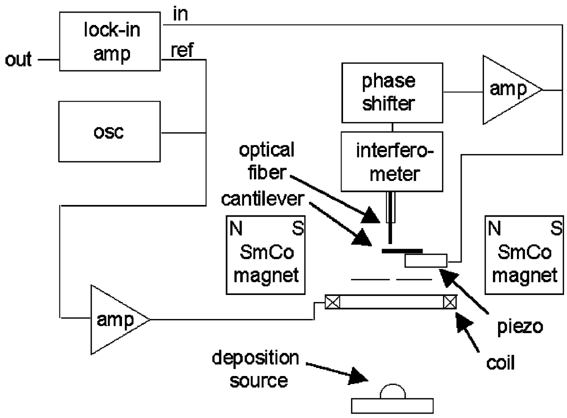

Fig. 1. Schematic diagram showing the components of a resonating torque microbalance (not to scale).

Fe films (99.9%) were deposited onto custom fabricated single-crystal silicon cantilevers. The cantilever geometry is shown in Fig. 2. Depositions were done in a diffusion-pumped vacuum chamber with a liquid nitrogen cold trap. The cantilever was masked by a 100 μm thick mica sheet positioned 0.5 mm from the cantilever surface. The mica sheet had a 1 mm diameter hole in it exposing only the cantilever substrate to the deposition source. In addition, the cleaved end of the optic-fiber was positioned a few micrometers from the back substrate near the center. The background pressure during deposition was 2.66×10^{-4} Pa. The films were evaporated from alumina coated tungsten boats at a deposition rate ranging from 0.1 to 1 nm/s. Film thickness was measured with a commercial quartz crystal thickness monitor with a precision of 0.1 nm.

The torque on a uniformly magnetized thin film with a strong in-plane anisotropy is $T_M = \mu_0|m \times H_T| = \mu_0 m H_T$, assuming a 90° angle between H_T and the magnetic moment m of the film. For the paddle configuration described in Fig. 2 we assume that the torque on the film acts as a bending moment concentrated at the end of the cantilever spring neglecting any bending of the cantilever substrate. The displacement z is therefore [5]

$$z_r = \frac{6T_M l_c^2}{E w_c t_c^3}, \quad (1)$$

$$= \frac{6\mu_0 m H_T l_c^2}{E w_c t_c^3}, \quad (2)$$

$$= \frac{6\mu_0 M_s t_f a_f H_T l_c^2}{E w_c t_c^3}. \quad (3)$$

With the parameters defined in Table 1 (note that the magnetic torque is defined to be $T = \mu_0 M_s a_f t_f H_T$). At resonance there is a Q enhancement [6] of $z_r = Q \times z$. Fig. 3 shows the resonance peak for the cantilever for magnetic, piezo, and thermal excitation of the cantilever with a 25 nm thick Fe film deposited onto the substrate. The thermal peak is the correlated Brownian motion of the cantilever detected by the lock-in amplifier. From these data we determine the resonant frequency to be 3276 Hz and the Q to be at least 1500. For a 25 nm thick Fe film given the experimental parameters shown in Table 1 we find that $z_r = 120$ nm rms. This number is in reasonable agreement with our experimental measurement $z_r = 100$ nm rms shown in Fig. 3 given uncertainties regarding the cantilever geometry (particularly thickness).

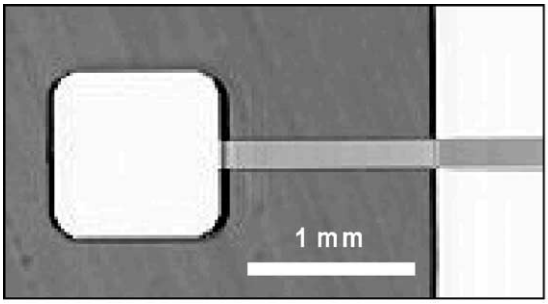

Fig. 2. Photomicrograph of a custom fabricated Si paddle cantilever for in situ torque magnetometer measurements. Films are deposited onto the substrate only. Both the substrate and spring are 20 μm thick.

Table 1
Experimental parameters

Symbol	Definition	Value
μ_0	Permeability of free space	$4\pi \times 10^{-7}\ H/m$
M_s	Saturation magnetization	1×10^6 A/m (Ref. [1])
a_f	Magnetic film area	$\pi \times$ (0.5 mm)^2
H_T	Torque field	300 A/m rms
l_c	Cantilever length	1.2 mm
w_c	Cantilever width	200 μm
t_c	Cantilever thickness	28 μm
E	Young's modulus	1.79×10^{11} N/m^2
Q	Cantilever quality factor	1500

Fig. 4 shows results for a Fe film deposition. Notice that the moment of the film is nearly proportional to the thickness of the film over the course of the deposition. The deposition rate was varied over time from 0.7 to 0.5 nm/s and then back to 0.7 nm/s. Lower deposition rates had lower magnetic moments versus thickness slopes. Lower deposition rates lead to more water being included in the film and thus may decrease the bulk moment of the film. The average magnetic moment noise level corresponds to 0.2 nm Fe film thickness equivalent.

Similar data can be obtained without active feedback. In such cases the magnetic moment signal is dominated by small shifts in the cantilever resonance frequency

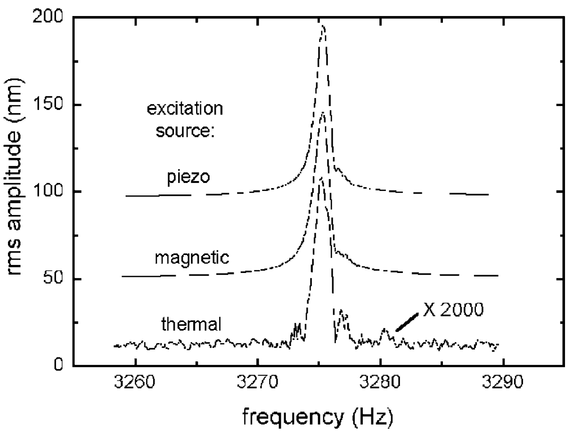

Fig. 3. rms amplitude versus frequency for the cantilever shown in Fig. 2. The cantilever response is shown for piezo and magnetic excitation. The thermal peak is due to the Brownian motion of the cantilever without external excitation (curves displaced for clarity).

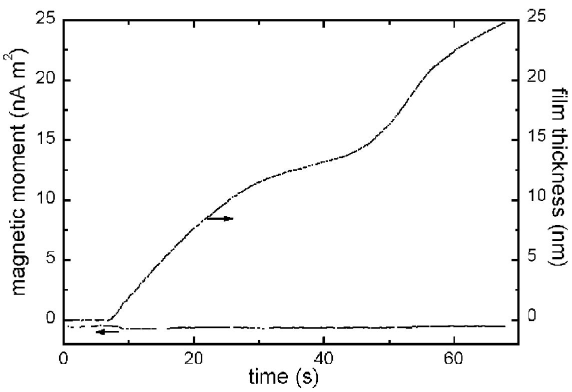

Fig. 4. Magnetic moment measured with the resonating torque microbalance and the thickness measured with a quartz crystal thickness monitor versus time during a Fe deposition (curves displaced for clarity).

caused by thermal and mass loading affects rather than changes in magnetic moment. These spurious affects are amplified when operating near the resonance of cantilever. To check that the active feedback mechanism is working properly, and that the mechanical torque supplied by the piezo is reacting to only the magnetic torque, we onto the Fe film (see Fig. 4). As expected, the magnetometer shows little response during the Cu deposition.

In principle, the fundamental noise source for these measurements is the Brownian motion of the cantilever, which can be expressed as an equivalent thermal noise per root hertz of [7]

$$z_{\min} = \sqrt{\frac{2k_B T}{\pi k_s Q f_0}}, \tag{4}$$

where k_s is the spring constant, Q is the mechanical quality factor, f_0 is the resonant frequency, and $k_B T$ is the thermal energy. Using the parameters in Table 1 we find that $z_{\min} = 1.2 \times 10^{-14}$ m. Given the Q enhancement this number agrees well with the thermal peak in Fig. 2 in a 1 Hz measurement bandwidth; $z_{\text{therm}} = Q z_{\min} = 0.013$ nm indicating that we are operating near the thermal limit of the cantilever. However, during deposition, a 60 Hz (plus harmonics), 20 A current is flowing through the evaporation boat. Apparently this current couples to the cantilever either by electromagnetic coupling or vibrational coupling at a level 10 times greater than the Brownian noise contribution. The noise level during deposition corresponds to a magnetic film thickness sensitivity of 0.2 nm as discussed above (see Fig. 3). If the Brownian motion limit can be maintained during deposition we expect that the magnetic thickness sensitivity would be 0.02 nm.

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